Performance Monitoring Protocol (QA/QC) for the Scanning Electron Microscope (SEM) / Energy Dispersive X-ray Spectrometer (EDS)

1 Scope

This document addresses the performance monitoring (QA/QC) of the Scanning Electron Microscope (SEM) / Energy Dispersive X-ray Spectrometer (EDS). This document applies to personnel using the associated instrument(s)/equipment in the following disciplines/categories of testing: Paint, explosives (chemistry), drug chemistry, and Chemistry Unit general physical and chemical analysis.

2 Principle

The SEM/EDS is utilized primarily to characterize the elemental composition of a material. Because this instrumentation is dependent upon a determination of the energy of detected X-rays, it is necessary to ensure the instrument is performing optimally for the intended analysis.

SEM can be utilized for morphological and metrological determinations; therefore image quality and measurement accuracy will need to be determined for these examinations. For routine analysis the magnification accuracy is sufficient. When a measurement is required with reportable accuracy, a calibrated measurement standard will be employed. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

3 Equipment/Materials/Reagents

a. Scanning Electron Microscope (SEM) – JEOL JSM 6510LV, JEOL JSM6610, TESCAN Vega 3 XMU (or equivalent)

b. Energy Dispersive Spectrometer (EDS) - EDAX (or equivalent)

c. Ruler, metric - Fisher Scientific (or equivalent)

d. Energy Dispersive X-ray processing software – EDAX Genesis (or equivalent)

e. Spectrum Library Identification and Classification Explorer (SLICE) software, xk, Inc (or equivalent)

g. Manganese (Mn) standard, polished (or equivalent)

h. Magnification standard - Geller MRS-3 SEM (or equivalent)

4 Standards and Controls

Prior to each use of the SEM/EDS, a determination of the ability to perform elemental identification will be made by confirming system energy calibration using the X-ray lines of the pure element standard manganese (Mn).

5 Calibration

Calibration of the EDS will only be performed if the X-ray lines are shifted from their expected positions in the spectrum of the pure element standard manganese (Mn) by more than 30 eV. This procedure will be performed by properly trained personnel.

6 Sampling or Sample Selection

Not applicable.

7 Procedures

7.1 Daily Checks

a. Collect a spectrum from the pure element standard manganese (Mn).

b. Recall verification spectrum of previous pure element.

c. Compare spectra. Observe and compare peak shape, peak width, high to low energy peak ratio, shape of background, peak artifacts, and system peaks.

d. If the peaks observed are shifted from their previously established positions by more than 30 eV, then contact appropriate instrument support personnel.

e. Print spectrum, initial, date and insert into appropriate section of the QA/QC log.

7.2 Magnification Standardization

Magnification standardization will be performed, as follows, when a value will be reported. Additional steps may need to be taken in order to report a value (e.g., measurement uncertainty calculations, traceability considerations).

a. Place the Geller MRS-3 SEM magnification standard into SEM chamber and evacuate.

b. Set specimen tilt to 0 degrees (perpendicular to the electron beam).

c. Adjust the instrumental conditions to be similar to those used to image the object

from which an accurate measurement is required.

d. Bring the Geller MRS-3 SEM magnification standard into focus using stage Z.

e. Take one digital image of an appropriate pattern on the Geller standard. Different pattern selections are available depending on the magnification selected. Examples are provided below:

Pattern	Spacing
50X	largest bar plus space = 0.5 mm
100X	largest bar plus space = 0.5 mm
1,000X	middle bar plus space = 50 μm
10,000X	smallest bar plus space = 2 μm

f. Using a ruler, measure the features in the digital images and calculate the actual magnification.

g. Calculate % magnification error, and apply the correction to the measurement from the structure of interest.

h. Record results in QA/QC log.

i. Once a magnification error has been determined for a specific set of analytical conditions, error correction can be applied to any subsequent measurements obtained under those conditions.

8 Instrumental Conditions

8.1 Imaging

Detector type (e.g. secondary or backscatter) and values for accelerating (high) voltage, working distance, spot size, beam intensity, stigmation, focus, brightness, and contrast are established at the operator's discretion based on image quality desired.

8.2 Magnification

Instrumental conditions will be the same as those required to image a material of interest from which a precise measurement is required.

8.3 EDS Detector

Detector response: Mn
Beam voltage: 25KV

Working distance and beam intensity/spot size will be set at the operator's discretion.

9 Calculations

magnification = (image dimension)/(object dimension)
% magnification error =
[(displayed magnification – measured magnification)/measured magnification] x 100

10 Measurement Uncertainty

Not applicable.

11 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of the SEM and/or EDS.

12 Decision Criteria

a. Detector response:
In order for the instrument to be considered in good operating condition, the manganese spectrum must appear generally similar to the previously collected manganese spectra. The spectrum should exhibit a similar high-to-low energy peak ratio, Gaussian peak shape, a minimum SNR of 3:1, and the absence of any significant spectral artifacts. Changes in the low-to-high peak intensity ratio may indicate accumulation of ice on the crystal face.

b. Energy characterization:
If the measured peak centroid energy is more than 30eV from the theoretical average Mn Kα peak energy of 5.895 keV, a detector calibration will be performed in accordance with the manufacturer's recommendations.

c. If all requirements are within specification, prepare documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, the IOSS Manager or appropriate instrument support personnel will determine the corrective maintenance to be performed.

13 Safety

General precautions common to electron microscope laboratories include: Venting of P-10 gas, venting or filtering of roughing pump discharge, and avoidance of direct exposure to beryllium metal. Under normal operator conditions the instrument poses no known hazards.

Use universal precautions when handling potentially biohazardous materials. Take standard

precautions for the handling of chemicals and sharp cutting instruments. Refer to the *FBI Laboratory Safety Manual* and appropriate SDSs for additional required practices and precautions.

14 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

Goldestein, Newbury, Echlin, Joy, Romig, Lyman, Fiori, Lifshin, *Scanning Electron Microscopy and X-ray Microanalysis,* Second Edition, Plenum Press, 1992.

"General Instrument Maintenance Protocol" (Inst 001) *Instrument Operation and Systems Support SOP Manual.*

FBI Laboratory Safety Manual.

Rev. #	Issue Date	History
2	12/05/11	Removed dead time requirement from section 8.3.
3	10/04/18	Updated Section 1 Scope to include disciplines/categories of testing. Added JEOL JSM6610 to Section 3 a. Updated heading in Section 6. Removed specific software titles from Section 7.1 b. Removed requirement to overlay spectra in Section 7.1 e. Added 'appropriate instrument support personnel' to Sections 7.1 d and 12 c. Added additional steps for reporting to Section 7.2. Added detector types in Section 8.1. Added 'EDS' to Section 8.3 title. Added theoretical value in Section 12 b. Removed outdated cryogen hazard from Section 13. Updated 'Instrument Operation and Systems Support' in Section 14 and header.

Approval

Redacted - Signatures on File

Drug Chemistry/
General Chemistry
Technical Leader: Date: 09/28/2018

Metallurgy
Technical Leader: Date: 09/28/2018

Paints and Polymers
Technical Leader: 09/28/2018

Explosives (Chemistry)
Technical Leader: Date: 09/28/2018

IOSS Manager: Date: 09/28/2018

Chemistry Unit Chief: Date: 09/28/2018

QA Approval

Quality Manager: Date: 09/28/2018